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EN-84-12

AUTOMATED IDENTIFICATION, SIZING AND COUNTING OF PARTICULATE CONTAMINATION FOUND IN HYDRAULIC RECOIL SYSTEMS

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AUGUST 1984

U.S. ARMY
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TECHNICAL REPORT

ENGINEERING DIRECTORATE
ROCK ISLAND ARSENAL
ROCK ISLAND, ILLINOIS 61299-5000

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1. REPORT NUMBER EN-84-12	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) AUTOMATED IDENTIFICATION, SIZING AND COUNTING OF PARTICULATE CONTAMINATION FOUND IN HYDRAULIC RECOIL SYSTEMS		5. TYPE OF REPORT & PERIOD COVERED Technical Report 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) W. D. McHenry and L. A. Post		8. CONTRACT OR GRANT NUMBER(s) DAAA08-80-F-0092
9. PERFORMING ORGANIZATION NAME AND ADDRESS Commander, Rock Island Arsenal ATTN: SMCRI-ENM-E Rock Island, IL 61299-5000		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 5397.OM.6350
11. CONTROLLING OFFICE NAME AND ADDRESS Commander, Rock Island Arsenal ATTN: SMCRI-EN Rock Island, IL 61299-5000		12. REPORT DATE August 1984 13. NUMBER OF PAGES 57
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)
Approved for public release, distribution unlimited.

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

1. Hydraulic Recoil Mechanism
2. Scanning Electron Microscope
3. Secondary Electrons
4. Backscattered Electrons
5. Characteristic X-rays
6. Energy Dispersive Spectrometer
7. LeMont Image Analysis System (B-10)
8. Automated Particle Counting System
9. Data Save

(See reverse)

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Procedures for the automated identification, sizing, and counting of particulate contamination in hydraulic recoil systems have been developed. The automated system can provide meaningful, accurate data in various formats and a dedicated Automated Particle Analysis system has the potential to provide results quickly to meet production needs on an unattended operating basis.

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

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Block No. 19

KEY WORDS
(continued)

10. Chemical Definition File
11. Diameter
12. Gridameter
13. Relative Intensity Classification

FOREWORD

To develop an automated system to size, characterize, and count particles contained in artillery recoil mechanism hydraulic fluids.

This project has been accomplished as part of the US Army Materials Testing Technology Program, which has for its objective the timely establishment of testing techniques, procedures or prototype equipment (in mechanical, chemical, or nondestructive testing) to insure efficient inspection methods for materiel or material procured or maintained by DARCOM.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF FIGURES (Con't)

1.0 INTRODUCTION

1.1 Background.

Rock Island Arsenal (RIA) manufactures or rebuilds several different hydraulic recoil mechanisms for artillery weapons. The contamination level in the hydraulic fluid in the mechanisms is checked before their release for shipment. Limits on the number, size, and character of the particles permitted for each mechanism are given in Table I.

Federal Test Methods Standard No. 791b, Method 3009.2, "Solid-Particle Contamination in Hydraulic Fluids," delineates procedures used to optically determine the size and number of particles filtered from the hydraulic fluids of interest. The procedures were extended because of the drawing requirements to include characterization of particles to determine whether they are metallic (ferrous or non-ferrous) or non-metallic (abrasive, non-abrasive, or lint and fiber).

1.2 Optical Particle Analysis Method.

The present method of inspection is to drain a stipulated amount of hydraulic fluid from a newly assembled mechanism immediately after it has been exercised or gymnasticated. This fluid is filtered through a fibrous filter paper and the debris remaining on the filter paper is examined with an optical microscope. A calibrated micrometer eyepiece in the microscope is used to determine particle size. A magnet, a probe, and appearance are used to qualitatively characterize the particles of interest. These manual methods are time consuming and subjective. The subjectivity is influenced by the capability, training, and of great importance — the amount of eye fatigue experienced by the person performing the test.

1.3 Automated Particle Analysis Method.

This Manufacturing Testing Technology (MTT) project was initiated to determine the feasibility of automating the particle counting procedure with the use of a scanning electron microscope (SEM). This investigation consisted of four phases:

a. Phase 1 - Calibration and Preliminary Testing of Automated System.

b. Phase 2 - Development of Sample Preparation Techniques.

c. Phase 3 - Determination of Operating Parameters.

d. Phase 4 - Collection and Analysis of Data to Determine Feasibility of Using the Automated System.

TABLE I

Particle Size Criteria for Mechanisms and Gun Mounts Produced at RIA (Micrometers)

	M1 [1]	M45 [2]	M140 [3]	M174 [4]	M178 [5]
Oil Sample Size	1 Pint	2 oz.	1 Pint	1 Pint	1 Pint (From each Recoil Cylinder)
Metallic - Ferrous	600	400	Max. of 50 particles between 100 and 300. Max. of 5 particles between 301 and 400. None over 400.	Max. of 2 particles between 40 and 200. None over 200.	Max. of 2 particles between 40 and 200. None over 200.
Metallic - Nonferrous	600	400	Max. of 20 particles between 200 and 400. None over 400.	Max. of 2 particles between 200 and 400. None over 400.	Max. of 2 particles between 200 and 400. None over 400.
Nonmetallic - Abrasive	600	400	Max. of 100 particles between 200 and 3000. None over 3000.	Max. of 2 particles between 200 and 400. None over 400.	Max. of 2 particles between 200 and 400. None over 400.
Nonmetallic - Nonabrasive	600	400	Max. of 100 particles between 200 and 3000. None over 300.	Max. of 3 particles between 400 and 600. None over 600.	No limit, but monitor closely to assure minimal quantity.
Nonmetallic - Lint and Fiber	600	400	Max. of 100 particles between 200 and 3000. None over 3000.	Max. of 4 particles between 600 and 60 and 3000 by 200. None over 3000 by 200.	Max. of 8 particles between 600 by 60 and 3000 by 200. None over 3000 by 200.

[1] M1 Gun Mount: Design Specification Requirements - Drawing 12274292, Revision J, 18 Jun 82, US Army Armament Research and Development Command. Maximum particle sizes.
[2] M45 Recoil Mechanism: Mechanism, Recoil, 155mm Howitzer: M45, MIL-M-45986(AR), Amendment 2, 10 May 82, Paragraph 3.3.6.2. Maximum particle sizes.
[3] M140 Gun Mount: Note 10, Drawing 12000890, Revision C, 23 Jul 79, US Army Armament Research and Development Command.
[4] M174 Gun Mount: Disposition Form, Subject: M158 Mount-New Manufacture-Hydraulic Oil Contamination Criteria, SARRI-LA to SARRI-QP, and SARRI-FE, 7 May 1975 (Note: The M158 Mount was redesignated the M174 Gun Mount).
[5] M178 Gun Mount: Note 3.4.1, Drawing 12012354, 7 Feb 79, Rock Island Arsenal.

An SEM is similar to a closed circuit television system in that the portion of the sample being observed is scanned in a raster pattern by a moving beam of electrons. The interaction of the electron beam with the sample produces several effects, three of which were considered for use in this project.

a. Secondary electrons emitted from the surface of the sample. These low energy electrons carry much information related to the topography of the surface being observed and are used during the preliminary observations of the sample.

b. Backscattered electrons from the sample. The number of electrons rebounding varies directly with increasing atomic number of the material being observed. This property can be used within limits to separate the signals from higher and lower atomic weight materials.

c. Emission of X-rays with energies characteristic of the elements being scanned. The SEM spectrometer can be used to determine which elements with atomic numbers 10 and higher are present in the sample. This includes elements that are common contaminates such as iron, chromium, copper, nickel, and aluminum.

Signals produced by detectors for each of these three effects are processed electronically and are presented on a video screen. Magnification obtained is inversely proportional to the ratio of the raster size on the sample to the size of the viewing screen. All functions of the SEM are digitally controlled and all information produced can be presented in digital form. Thus, operation of the SEM and processing of the data produced can be done by a dedicated computer that is part of the RIA microscope, and the information gained from these three effects is free of the subjective factors present in the optical counting method.

Of the several particle counting systems currently available commercially, the LeMont system was chosen for this project because of its:

a. Ability to characterize, size, and count particles.

b. Compatability with equipment already on hand. LeMont uses the same computer which was already a part of the automated X-ray wavelength spectrometer on the RIA SEM. Also, LeMont interfaces easily with energy dispersive X-ray spectrometers of the type available on the RIA instrument.

c. Flexibility. The LeMont system is mainly software oriented. Once the basic hardware is installed, all functions and operations are controlled by the software. Modifications to the programs caused by changes in requirements are easily accomplished. The addition of new instrumentation requires only a change or addition to the program.

The LeMont Image Analysis System (B-10) as it is configured at RIA interfaces a research model ETEC Corporation Scanning Electron Microscope and a KEVEX Corporation 7000 Energy Dispersive Spectrometer to a Perkin-Elmer 16 bit Interdata Computer. An overall view of the instrumentation is shown in Figure 1. By computer control of the electron beam in the SEM, the LeMont software measures the physical dimensions of the particles and collects X-ray data on each particle.

Physical dimensions are determined by stepping the beam across the sample surface until it encounters a particle of higher atomic weight than that of the filter. The backscattered electron signal then goes above the selected threshold level indicating the presence of a particle. When searching for a particle the step size is set so that most of the small (less than 40 micrometer) particles are stepped over and are ignored.

While dimensioning or measuring a particle, the step size is reduced so that accurate measurements may be made. Either of two algorithms can be used for examining the particle. The first, "Diameter," constructs horizontal, vertical, and diagonal measurements about the centroid of the particle; width is the minimum diameter and length is the maximum diameter (Figure 2). The second algorithm is a grid measuring program, "Gridameter," which constructs a horizontal and vertical grid over the surface of the particle and calculates width, length, and centroid in several different ways depending on the shape of the particle. Gridameter can be used to examine long or crossed fibers, conglomerates, and particles with arms or voids (Figure 3). Chemical data for each particle is obtained by placing the electron beam at the particle centroid and collecting the X-ray data for a short time (two to five seconds). By measuring the relative amounts of elements detected, the particle is categorized into one of the groups shown in Table I.

The LeMont programs also control motions of the sample within the SEM chamber, i.e., translation in the X direction (the sample moves horizontally on the viewing screen) and rotation. These two parameters allow the essentially flat and smooth sample to be moved without the area to be observed going out of focus.

In LeMont terminology, the actual area of the sample being covered by the electron beam raster is called a "frame." The software provides several different combinations of the X direction and rotate motions to cover samples of various configurations. RIA samples are flat and circular, thus the chosen motion starts at the center of the sample, translates in X, rotates 360 degrees at 60 degree intervals, translates again, rotates again at smaller intervals and repeats once more giving the pattern of 37 frames to cover the sample (Figure 4).

FIGURE 1

THE RIA SCANNING ELECTRON MICROSCOPE AND PERIPHERAL PARTICLE COUNTING AND X-RAY EQUIPMENT.

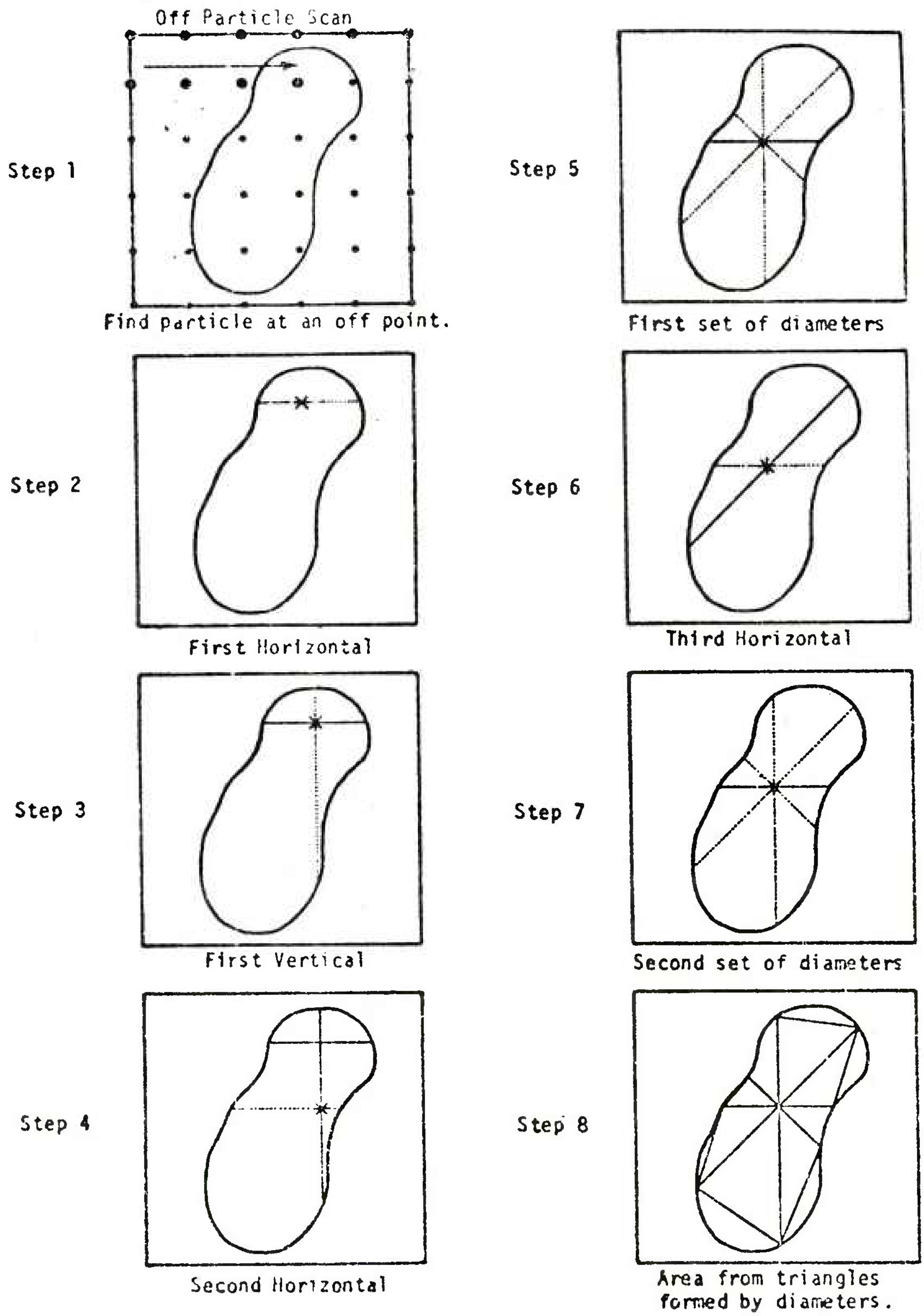


FIGURE 2

DIAMETER ANALYSIS.

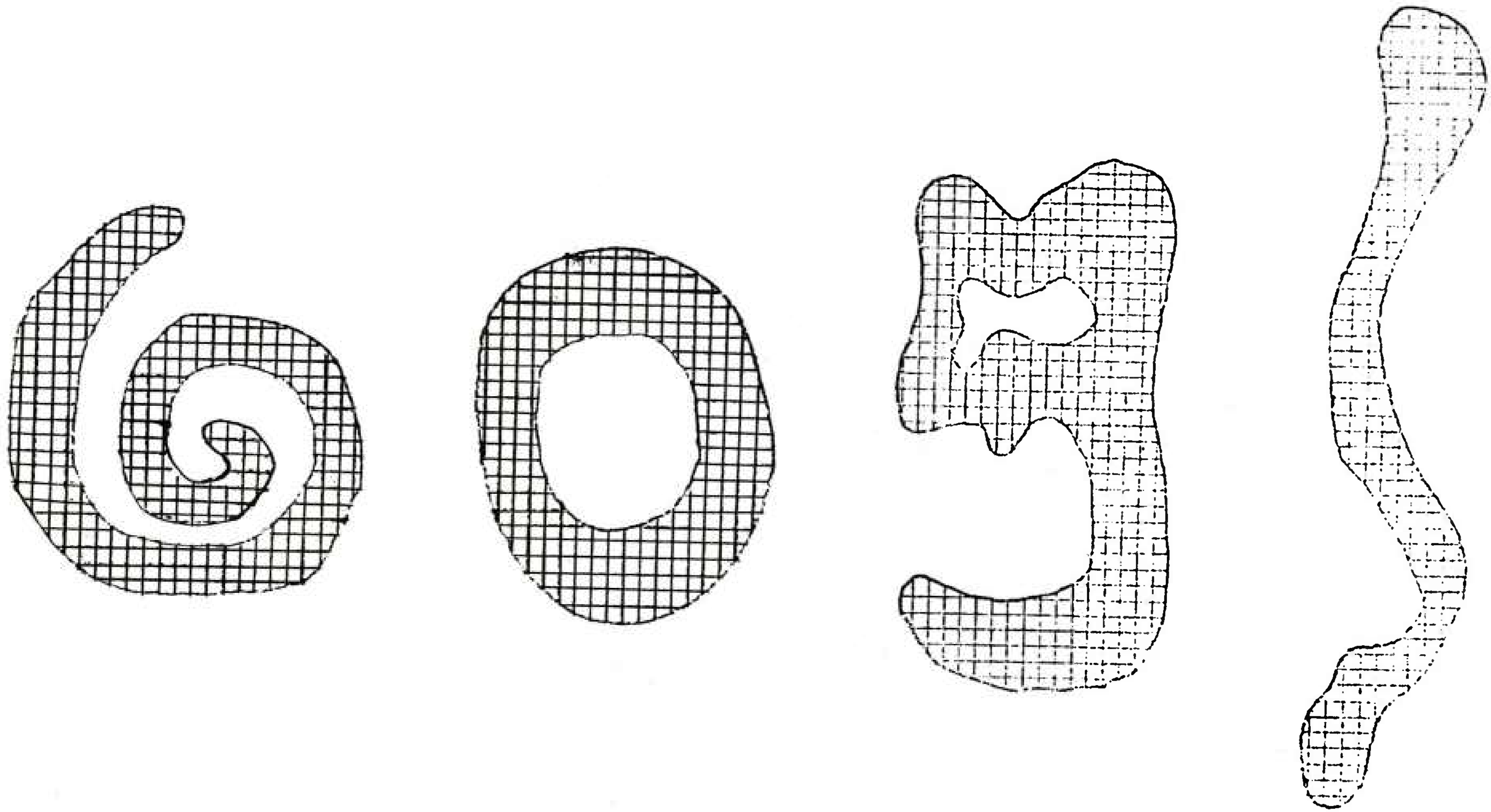


FIGURE 3

GRIDAMETER ANALYSIS.

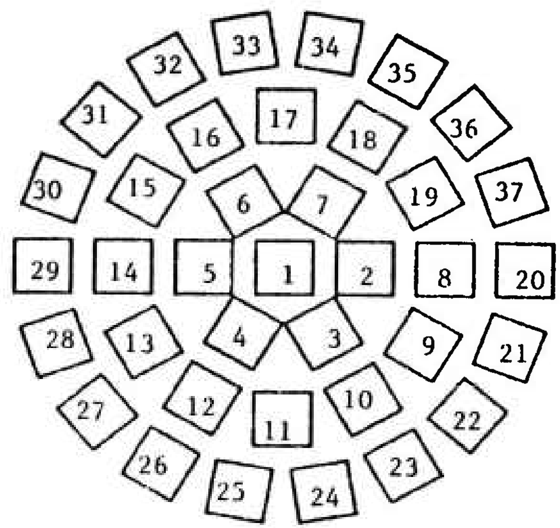


FIGURE 4

37 FRAME PATTERN.

1.4 Contracts with LeMont Scientific.

Contract DAAA08-78-R-0078. The purpose of this contract was to purchase the LeMont hardware and software required for the particle counting project. The hardware included:

a. Digital Scan Generator (positions the electron beam in the SEM).

b. Threshold Selector (determines which signals will be used to determine particle size).

c. Eight Channel Analyzer (determines the presence of seven elements plus background from the signal received from the energy dispersive X-ray detector). This analyzer was used with the Nuclear Diodes X-ray system on the SEM at the time of this contract.

d. Required power supplies, instrument rack, computer interface, and cables.

The software included the required computer languages, operating and editing systems. Also, provided were: (a) the LeMont programs to control the SEM, (b) programs to tabulate the particle size, numbers, and composition, (c) programs for a customized printout in an RIA format, and (d) programs to retrieve and re-examine under different parameters the original data which is stored on magnetic disks.

The contract also included installation, calibration, and training and was completed in October 1978.

Contract DAAA08-80-F-0092. This contract with LeMont Scientific developed the means of sample preparation, determined proper operating parameters for the system, and provided training for personnel at RIA on all procedures developed. This contract also included the purchase of additional software for chemical classification using up to 32 elements. The increased chemical classification became possible when the original Nuclear Diodes X-ray spectrometer failed and was replaced by a much improved, state-of-the-art KEVEX system. Procurement of the new X-ray system caused an eight month delay in the project. This LeMont contract was completed in July 1980.

Contract DAAA08-80-F-0093. This contract was for the automation of the SEM sample handling stage so that the sample being examined can be positioned by the computer. This places the entire analysis under computer control. The contract was completed in December 1980.

Contract DAAA08-82-M-1462. This contract was for the purchase of LeMont's latest particle counting program, "Gridameter," which incorporates the use of a simplified operating system and a more sophisticated algorithm. The contract included the conversion of the special RIA programs for use with Gridameter. The contract was completed in December 1982.

2.0 PROCEDURE

2.1 Calibration and Preliminary Testing

Calibration.

a. An aperture from the electron column of the SEM was used as the standard for the calibration of the LeMont system. A nominal 150 micrometer diameter aperture was optically measured using a microscope equipped with a micrometer stage and digital readout. The arithmetic mean of ten measurements (five each in the X and Y direction) was 157 micrometers. This 157 micrometer "hole" was made to look like a particle to the LeMont system by inverting the polarity of the image signal (Figure 5). X and Y coordinates were adjusted until the major and minor diameters of the "particle" as determined by the automated system were within plus or minus 5 micrometers ($\pm$ 3%) of the 157 micrometer value. Table II gives the data from ten consecutive measurements of the aperture as determined by the automated system after calibration.

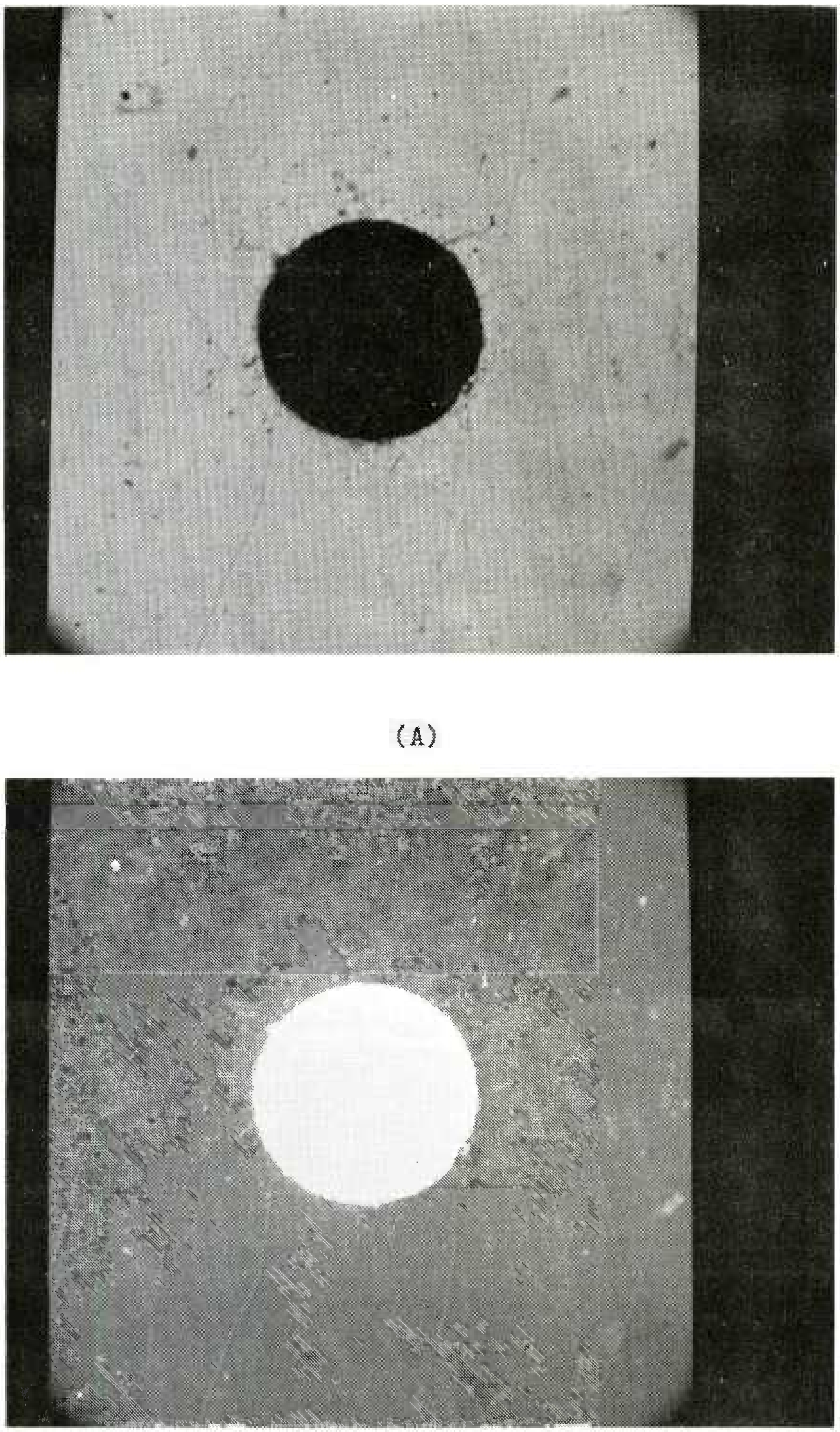
b. A multielement microprobe standard was used to determine the optimum energy ranges (windows) used to separate the X-rays of one element from another in the Kevex Energy Dispersive spectrometer. The elements chosen, their X-ray energies and thus the channels included in the windows are shown in Table III and Figure 6. The "element" designated MS In Table III occurs at the portion of the X-ray spectrum where the molybdenum L alpha X-ray and sulfur K alpha X-ray energies are very similar (2.293 and 2.307 KEV respectively) and cannot be resolved. One of the materials used as a lubricant in some recoil mechanisms is molybdenum disulphide (MoS_2), hence the symbol MS was assigned to this region.

c. Overall calibration of the EDS used the radiation from aluminum bronze. This allowed the simultaneous accumulation of X-rays from aluminum and copper (1.486 and 8.027 KEV, respectively) for fixing both ends of the 0 to 10 KEV spectrum.

Preliminary Testing.

a. A copper electron microscope specimen grid with rectangular holes was affixed to an SEM specimen stub with Aquadag. Aquadag is an electrically conductive "glue" used in SEM work that consists of a colloidal suspension of carbon in isopropyl alcohol. Again by inverting the polarity of the SEM signal, the holes were made to look like rectangular particles to the LeMont System.

b. A group of eight rectangular holes (identified by granules on the surface of the grid) were measured with their major axes oriented at 0, 45, and 90 degrees to the horizontal axis of the SEM (Figures 7, 8, and 9). The eight holes are labeled A thru H in each of the figures.



(A)

(B)

FIGURE 5

APERTURE USED TO CALIBRATE SYSTEM.

(A) APERTURE IN NORMAL MODE.

(B) APERTURE IN INVERTED MODE TO MAKE IT APPEAR AS A BRIGHT PARTICLE.

TABLE II

Ten Measurements of 157 Micrometer Aperture

Run Number	Width	Length
1	156.88	160.71
2	156.88	160.73
3	156.88	160.69
4	156.65	160.71
5	156.73	160.57
6	156.73	160.57
7	156.88	160.62
8	156.65	160.56
9	156.65	160.41
10	156.65	160.41
$\bar{X}$	156.76	160.60
s	0.1094	0.1179
V (%)	0.069	0.073

TABLE III

Energy Ranges of LeMont Windows

CHEMISTRY DEFINITION FILE: M 45 NOV. 1982
ELEMENT ASSIGNMENT TO XRAY ANALYZERS

ELE.ID	HEX.TYPE	NAME	PRESET INTEGRAL	: XRAY ANALYZER :
1	80000000	MG	0.00E 00	EDS ENERGY 1.16 - 1.34KEV
2	40000000	AL	0.00E 00	EDS ENERGY 1.40 - 1.56KEV
3	20000000	SI	0.00E 00	EDS ENERGY 1.64 - 1.82KEV
4	10000000	P	0.00E 00	EDS ENERGY 1.94 - 2.10KEV
5	08000000	MS	0.00E 00	EDS ENERGY 2.20 - 2.44KEV
6	04000000	CL	0.00E 00	EDS ENERGY 2.52 - 2.72KEV
7	02000000	CD	0.00E 00	EDS ENERGY 3.04 - 3.22KEV
8	01000000	SN	0.00E 00	EDS ENERGY 3.34 - 3.56KEV
9	00800000	TI	0.00E 00	EDS ENERGY 4.40 - 4.62KEV
10	00400000	V	0.00E 00	EDS ENERGY 4.82 - 5.08KEV
11	00200000	CR	0.00E 00	EDS ENERGY 5.28 - 5.54KEV
12	00100000	MN	0.00E 00	EDS ENERGY 5.76 - 6.02KEV
13	00080000	FE	0.00E 00	EDS ENERGY 6.26 - 6.54KEV
14	00040000	NI	0.00E 00	EDS ENERGY 7.32 - 7.62KEV
15	00020000	CU	0.00E 00	EDS ENERGY 7.88 - 8.18KEV
16	00010000	ZN	0.00E 00	EDS ENERGY 8.52 - 8.76KEV

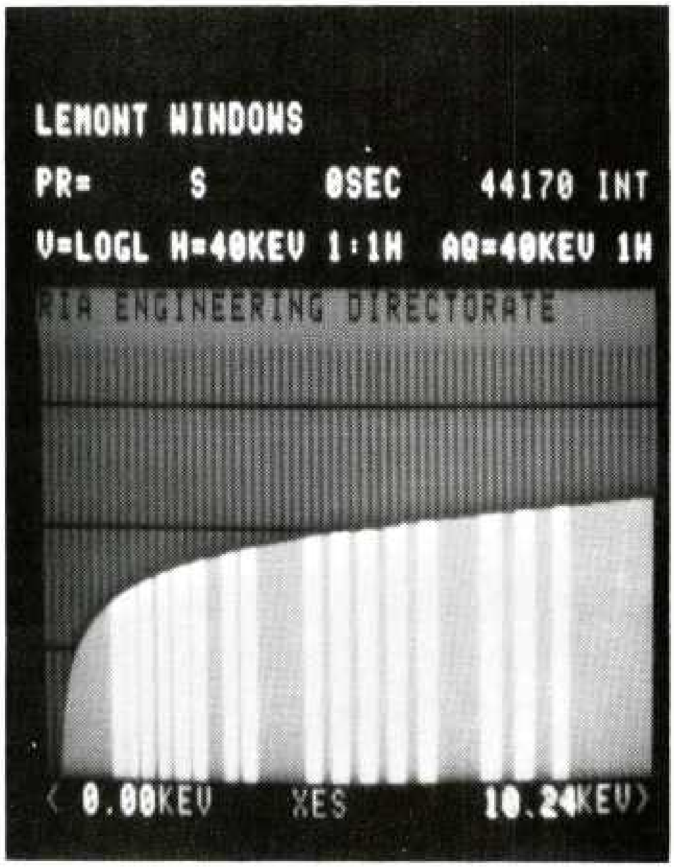


FIGURE 6

EDS SPECTRUM OF LEMONT WINDOWS.

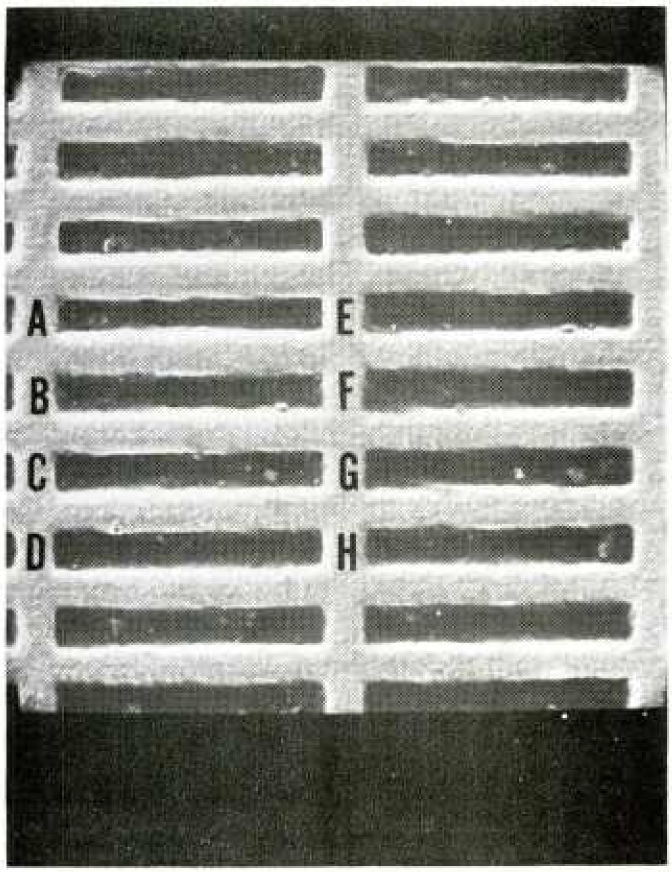


FIGURE 7

LENGTH OF GRID RECTANGULAR HOLES ORIENTED AT 0 DEGREES.

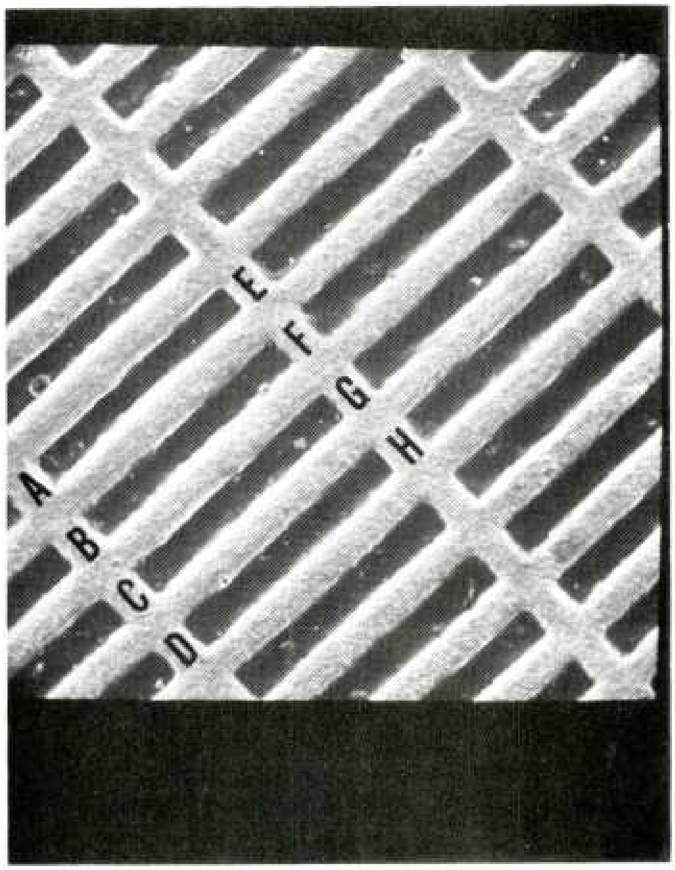


FIGURE 8

LENGTH OF GRID RECTANGULAR HOLES ORIENTED AT 45 DEGREES.

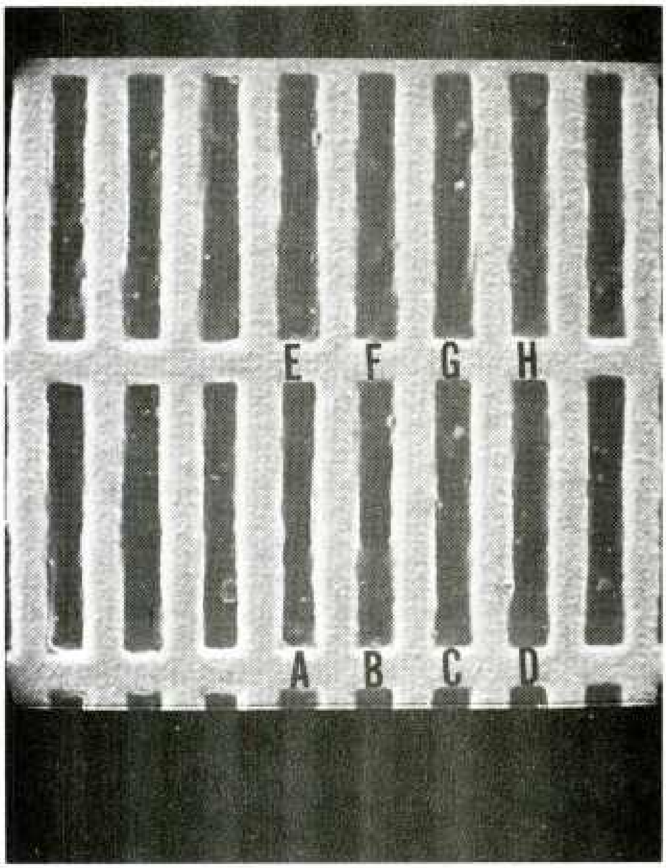


FIGURE 9

LENGTH OF GRID RECTANGULAR HOLES ORIENTED AT 90 DEGREES.

c. The arithmetic mean ($\overline{X}$) of the width and length measurements for the three orientations along with the standard deviation (s) and coefficient of variation (V) for the data is presented in Table IV. V is s divided by $\overline{X}$ times 100%, and gives the relative variability between groups of data in terms of percent. The reproducibility of measurement at the various orientations is well within the $\pm$ 3% criteria established for calibration. The reason for the slight decrease in the measured size of the holes when going from the 0 degrees to the 90 degrees orientation is attributed to electronic drift during the measurement period and the inability to exactly reproduce the electronic parameters after each set of measurements.

d. Based on the results presented above, the system was considered to be in calibration and to give reproducible results.

2.2 Sample Preparation.

LeMont Scientific developed a sample preparation technique to process oil samples for SEM analysis. Some of the procedure techniques were adopted as suggested but many required major changes to reduce the time consumed in sample preparation.

For sample preparation, polycarbonate membrane filters, were used as the particle collection surface. These filters are composed of elements of atomic number less than ten and so do not contribute any X-rays that can be detected by the KEVEX 7000 energy dispersive spectrometer (EDS). A pure carbon substrate on which to affix the filter following filtration was used. The pure carbon planchet does not generate any detectable X-rays when bombarded by electrons which penetrate the filter. A thin, conductive carbon coating was evaporated onto the nonconducting surface of the polycarbonate membrane to eliminate charging. (Charging is a buildup of electrons on a nonconducting surface which interferes with succeeding electrons, and consequently distorts the image.) The filter, carbon planchet, and planchet holder are shown in Figure 10.

Changes in the LeMont sample preparation technique included elimination of the twelve hour settling period for the oil sample. Also, instead of using a syringe to force the hydraulic fluid through the filter, a vacuum system was devised which pulled the fluid through the filter (Figure 11). This method proved to be faster and allows the filtering system to be washed down more easily.

Membrane filters with a fourteen micrometer pore size were used instead of the eight micrometer pore size recommended by LeMont. This change allows most of the particles which are under the specified size to pass through the filter and affords a significant reduction in time required for sample preparation.

The original method of holding the filter onto the carbon planchet was to use rings that screwed down on the planchet holder. This method caused the filter to wrinkle. The use of double-sided pressure sensitive

TABLE IV

Measurements of Rectangular Holes at 0, 45, and 90 Degrees

Orientation	A	B	C	D	E	F	G	H
				Width				
0	39.82	40.23	40.84	41.13	41.47	41.77	41.62	41.08
45	35.59	40.88	40.39	41.50	41.34	41.87	40.96	40.57
90	39.19	40.53	39.35	40.30	41.22	41.48	40.31	39.92
$\bar{X}$	39.53	40.55	40.19	40.98	41.34	41.71	40.96	40.52
s	0.319	0.325	0.764	0.615	0.125	0.203	0.655	0.581
V (%)	0.81	0.80	1.90	1.50	0.30	0.49	1.60	1.43
				Length				
0	289.30	296.79	290.41	289.84	291.86	294.95	291.67	292.63
45	281.40	283.10	284.06	281.59	284.67	284.54	284.30	283.55
90	274.81	276.80	281.05	280.52	278.75	277.49	279.15	276.47
$\bar{X}$	281.84	285.86	285.17	283.98	285.09	285.66	285.04	284.21
s	7.25	10.22	4.78	5.10	6.57	8.78	6.29	8.10
V (%)	2.57	3.58	1.68	1.80	2.30	3.07	2.21	2.85

(Hole Size, Micrometers)

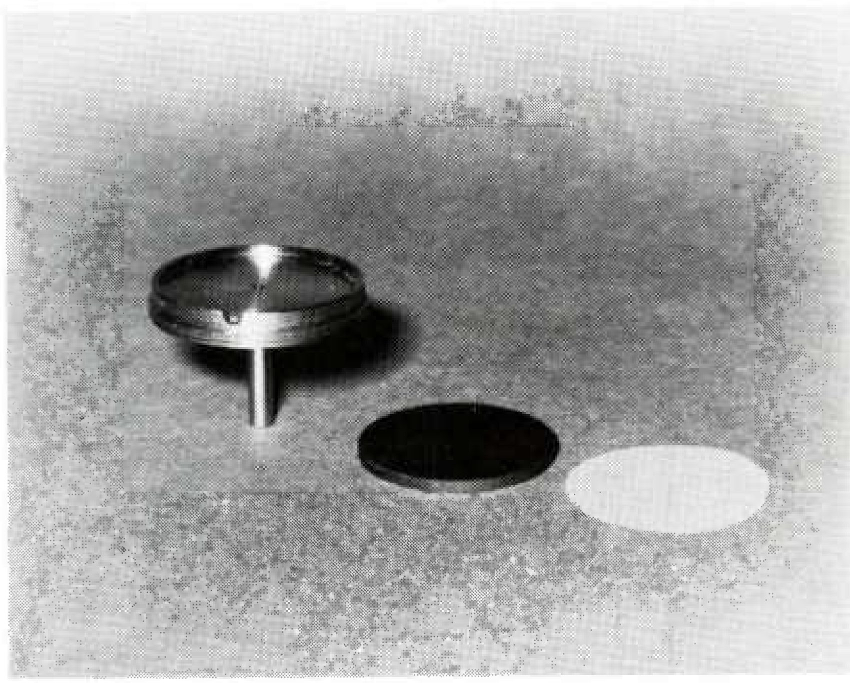


FIGURE 10

PLANCHET HOLDER, CARBON PLANCHET AND POLYCARBONATE MEMBRANE FILTER USED FOR AUTOMATED PARTICLE COUNTS.

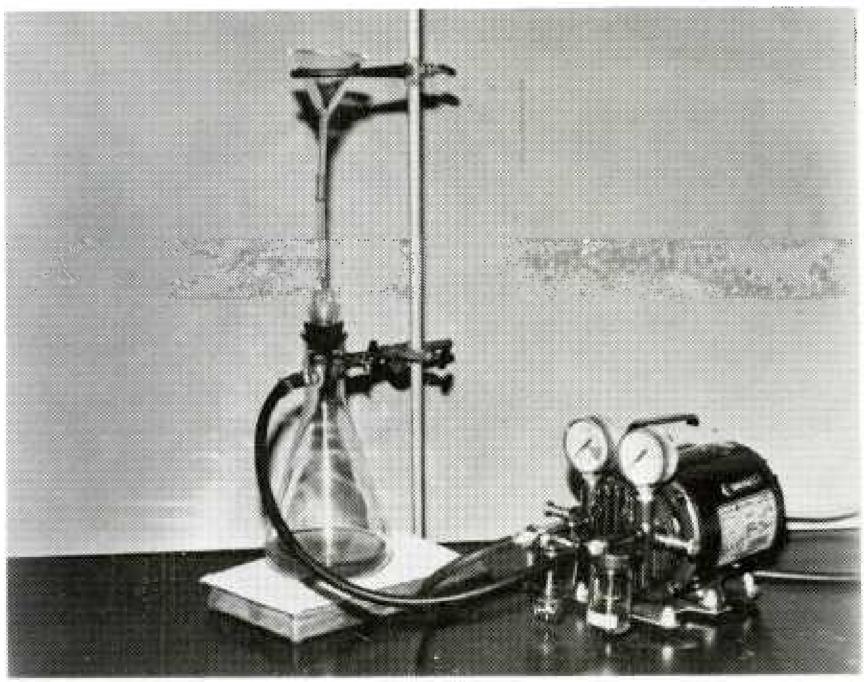


FIGURE 11

FILTERING APPARATUS USED FOR AUTOMATED PARTICLE COUNTS.

tape was investigated but this procedure was not satisfactory because it gave the operator only one opportunity to accurately position the filter on the planchet. Aquadag was applied to the planchet and worked successfully in holding the filter, but the Aquadag plugged the holes of the filter with carbon. These plugged holes generated the same backscatter signal as the filter (i.e., the holes had the same grey level intensity as the filter) and so the normal contrast of the black holes that is critical for setting electronic levels on the SEM was lost. Finally a solution of rubber cement dissolved in naphtha was applied to the carbon planchet. Use of this dissolved glue allowed easy positioning of the filter on the planchet with no wrinkles, and allowed the normal contrast of the black holes to be used for setting electronic levels on the SEM.

The carbon planchets are held in the planchet holder by friction alone to facilitate removal and storage of the samples following analysis. When the original samples are to be disposed of, the planchet and filter are soaked in naphtha while in an ultrasonic cleaner. This procedure loosens and removes the filter, and cleans the surface of the carbon planchet which can then be re-used.

The following procedure was used to prepare the M178, M174, M140, M45, and M1 oil samples. The sample size for the M45 is currently two ounces, and the sample size for each of the other mechanisms is currently one pint. The following steps were effective in preparing oil-free, conductive samples at RIA for analysis with the SEM.

a. Agitate the sample ultrasonically by placing the bottle containing it in an operating ultrasonic cleaner for ten minutes.

b. While the sample is in the ultrasonic cleaner, assemble the Swin-Lok Holder: (Figure 12).

(1) Unscrew the assembly ring and remove cap.

(2) Remove the base support grid and stretch the O-ring until it fits into the molded groove of the base.

(3) Replace the base support grid. The grid should hold the O-ring in place without pinching it.

(4) Select a Nuclepore membrane and using teflon-coated tweezers place the membrane on the base (shiny side toward the base). This is done because the oil flow through the assembled filter apparatus is from the base to the cap and the shiny side, being smoother, is preferred as the collection surface for microscopic analysis. Be sure the membrane completely covers the O-ring.

(5) Mate cap and base so the anti-twist tabs interlock.

(6) Screw assembly ring tightly onto base.

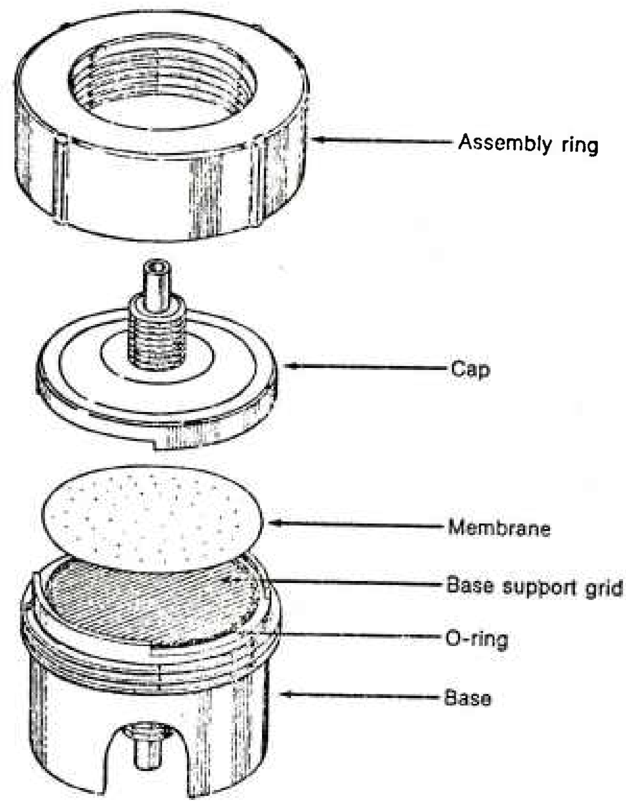


FIGURE 12

EXPLODED VIEW OF SWIN-LOK HOLDER.

(7) Insert assembled Swin-Lok Holder into vacuum system (Figure 11).

c. Take the oil sample out of the ultrasonic cleaner.

d. Adjust the vacuum so that minimum vacuum will be used to pull the fluid through the membrane filter (this alleviates the problem of particle pile-up around the perimeter of the filter), and then pour the fluid into the funnel of the vacuum system.

e. This step is optional and depends on how "dirty" the oil sample is. If the fluid begins to filter very slowly through the membrane and increasing the vacuum has little effect on the filtration rate; diluting the oil already in the funnel with clean petroleum ether will decrease the viscosity of the fluid and increase the filtration rate. Very dirty oil could require more than one membrane filter to completely filter the sample.

f. Carefully wash the oil residue out of the sample bottle with petroleum ether and filter this as well.

g. Wash the filter surface by filtering approximately 300 ml of clean petroleum ether through the filter. This step provides an oil-free surface on the filter and on the particles and fibers that are deposited on the filter. During the filtering operation avoid introducing air into the system because a blast of air sucked through the filter pushes the debris towards the edge of the filter depositing particles on top of each other.

h. Disassemble the Swin-Lok holder and transfer the membrane filter onto a carbon planchet which has been wetted with a coating of rubber cement dissolved in naphtha.

i. Place the sample under vacuum for a minimum of ten minutes to evaporate the solvents from the sample.

j. Finally, evaporate carbon onto the sample to form a conductive surface:

(1) The vacuum coating apparatus used to apply a conductive carbon coating to the sample is shown in Figure 13. Carbon is evaporated when a heavy electrical current flows through the junction of a 0.040 inch diameter pointed carbon rod and a flat face carbon rod. The evaporated carbon streams through the vacuum and is deposited on the sample.

(2) When the vacuum in the bell jar registers approximately 1x10E-04 Torr, evaporation can proceed. A current of 24-26 amperes should be maintained for 20-30 seconds to insure a good coating. Do not allow the current to exceed 26 amperes because sputtering of large carbon flakes will then occur. Slips of paper bent into a "v" shape should be placed in the bell jar near the samples. The shadowed area of this bent slip of

FIGURE 13

VACUUM EVAPORATOR.

paper remains uncoated and can be directly compared to the area on the paper slip which had been exposed during the coating process to give a rough idea of how heavy a carbon coating has been deposited on the sample.

Table V gives a breakdown of sample preparation time.

2.3 Operating Parameters

Sample and X-ray detector configuration and operating parameters for the RIA instruments were determined by LeMont during the on-site calibration portion of Contract DAAA09-78-R-0078. Subsequently, minor modifications were made as techniques were developed and "fine tuning" was required.

Final operating parameters for the SEM are:

a. Accelerating Voltage (for the electrons in the SEM column) - 20,000 volts.

b. Current through the sample - 1x10E-09 ampere.

c. Magnification - 40X. (A particle one millimeter long on the sample measures 40 millimeters long on a micrograph taken at this magnification.)

d. Frame size - 2.25 X 2.25 millimeters (mm). (The ETEC micrographs are 90 X 90mm, divided by 40X magnification gives the frame size of 2.25 X 2.25mm.)

e. Detector - Backscattered electron.

f. Working Distance - 14 millimeters. (Working distance is the distance between the final aperture in the electron column and the surface of the sample. Depth of the focus varies proportionally with working distance. A longer than the normal eight millimeter distance was chosen so that the small changes in the working distances between the surfaces of various types of particles would not affect focus.)

Parameters for the LeMont were not modified. A Low Pass filter (used to eliminate electronic noise) was set at 0.1 millisecond. A Dwell Time (the length of time the beam remains at each step or point) of 128 microseconds per point was used.

The computer controls most of the functions of the Kevex EDS; however, the LeMont program requires the multichannel analyzer be set at 20 electron volts per channel. A preset time of 3 seconds was determined to be sufficient time to collect the required X-ray information from each particle.

TABLE V

Summary of Sample Preparation Time

Time in ultrasonic cleaner - 10 Minutes.

Filter Time in Minutes: The time given is for filtering a single filter; if a sample requires more than one filter, multiply the time by the number of filters used.

M178	- 10
M174	- 10
M140	- 10
M45	- 5
M1	- 10

Sample Drying Time - 10 Minutes in vacuum oven.

Approximate pump down time (to draw vacuum of 1x10E-04 Torr). - 15 Minutes

Approximate carbon coating time: 20 - 30 seconds.

The total sample preparation time is 40 to 45 Minutes. In the course of preparing the oil samples for this project, time was saved by processing as many samples as possible at various steps. As many as four filters could be placed in the vacuum oven at one time for the ten minute outgassing period. Likewise, four filters have been successfully coated at one time.

The various brightness, contrast, and gain controls on the ETEC and LeMont systems were set such that signals from particles were of high enough intensity to go above the threshold and be processed while signals from the filter and substrate would not.

2.4 Test Plan.

A Test Plan was initiated to answer two questions:

a. What size and composition of particles would be found in different mechanisms?

b. How well does the LeMont system perform?

Duplicate samples were obtained from five of each of the five different mechanisms being produced at RIA. This amounted to 30 samples because the M178 Gun Mount has both an upper and lower cylinder and samples were obtained from both cylinders on each mechanism. All samples were filtered, observed optically (without using a probe), coated, and analyzed by the LeMont system.

Because the M1 Gun Mount came into production after LeMont provided the customized RIA programs, the M45 program (which is more restrictive, see Table I) was selected to count the M1 filters.

An M140 sample (serial number 16679, one of the first samples prepared) was used to determine the coverage of the filter surface obtained by the LeMont system. A full count of the sample (37 frames) was made with its initial frame at zero degrees rotation compared to the sample chamber. Eleven additional full counts were performed with the initial frame being rotated 30 degrees each time.

2.5 Description of Computer Printouts.

The LeMont system is very versatile in the manner in which results can be presented. They can be in the form of tables or histograms showing virtually any parameter measured versus another, i.e., chemical class vs size, width to length ratios, and percent of total area in each particle classification. Chemistry Definition Files can be specified which will instruct the computer programs to include only certain types or sizes of particles in the final printout. LeMont used this latter capability to prepare a customized printout in an RIA format based on requirements in effect at the time (contract 78-R-0078).

A format found to be very useful for this investigation is designated as a Particle by Particle Printout and has an automatic chemical classification in it. Particles are sorted into macro and subclasses by chemical composition in the following manner: A Relative Intensity Classification is performed on the X-ray data from the first particle detected. Percentages are assigned to each element detected based on the number of X-rays collected for that element compared with the total number

of X-rays collected from the particle. The program then creates a macro class consisting of those elements. The macro class is further subdivided based on the number of "levels" chosen. In this investigation, eight levels of twelve percent each were used so that the subdivisions were 0% to 12%, 13% to 25%, 26% to 37% on up to 88% to 99%. The percent of relative intensity determines into which level each detected element is placed.

When the second particle is detected and X-ray data are obtained the program searches to determine if the data will fit the previously created macro class. This will only happen if the same elements are detected. If there is not a match of the elements, a new macro class is created. If there is a match on the elements, the sub levels are checked. A difference in sub levels causes a second subclass to be created. As more particles are analyzed, they are placed into the proper macro and sub class, or, if need be, new classes are created for them until either no more particles are found or all available computer memory has been allocated in which case the count continues and particles are sized but not classified. The latter never occurred during the investigation.

After all data have been obtained from a particle and have been classified, a printout, as shown in Figure 14 is made.

```
               PARTICLE BY PARTICLE ANALYSIS FOR FRAME NO.   17

 -----  PARTICLE ----  -- POSITION -- ------------ MEASUREMENTS ------------
   S                            ANGLE   AREA   WIDTH/  WIDTH  LENGTH  LENGTH
ID-E CODE OR NAME   GRD  X    Y (DEG) (SQ.UM.) LENGTH  (UM.)   (UM.)   (UM.)
  1 SIFE             4  562 247   36. 1.48E 03  0.340 23.129  68.013 6.80E 01

    1170.XRAY CNTS SI FE
  2                18 82
------------------
```

FIGURE 14

INDIVIDUAL PARTICLE PRINTOUT.

(Explanation of headings and terms on next page)

EXPLANATION OF HEADINGS AND TERMS IN FIGURE 14

ID - Immediately below is the macro class number and four lines below is the subclass number.

S - An asterisk in this column indicates that this class has been selected to be used in histograms.

Code or Name - The chemical symbols of the elements in the macro class (in this case silicon and iron) are given.

GRD - The number of grid lines per off point used for sizing the particles.

X and Y - Location of particle centroid within the frame (based on 4096 divisions on each side of the frame).

Angle - The angle of the major diameter relative to the horizontal.

Area - The area covered by the particle calculated by the Gridameter program in units of square micrometers.

Width/Length - The ratio of the width divided by length, of the following width and length measurements.

Width - Length of minor diameter in micrometers.

Length - Length of major diameter in micrometers.

Length - This is an option column which can be used to present several parameters including perimeter, average diameter, and particle surface area (assuming the particle is an oblate spheroid.) None of the optional parameters was chosen for this investigation and length is repeated as the default option.

At the end of a complete run of 37 frames, the program prints out a summary for the Relative Intensity Chemical Classification as illustrated in Figure 15. Note that the particle described in Figure 14 is included as one of the two particles in Macro Class 1, sub Class 2, although one particle cannot be distinguished from another in the summary.

```
RELATIVE INTENSITY CHEMICAL CLASSIFICATION SUMMARY FOR

SAMPLE ID: M140-S11679-NEW PBGD W/AUTOCL OF CDVI-0 0535
============================================================================
MACRO CLASS   1 : SI FE
                %S 11 89
POP. =        4
POP.%=  28.57  AREA %=  32.51  AREA*XRAY %=  65.00  AVG XRAY CNT =      2324.
PARTICLES PER SQCM.= 2.32E 00  AVG.AREA = 1.60E 03  (SQ.UM.) SIGMA= 5.24E 02
----------------- SI FE
*SUB CLASS    1 UL 12 99
                LL  0 88
POP. =        2
POP.%=  50.00  AREA %=  41.36  AREA*XRAY %=  64.51  AVG XRAY CNT =      3634.
PARTICLES PER SQCM.= 1.16E 00  AVG.AREA = 1.32E 03  (SQ.UM.) SIGMA= 4.26E 02
----------------- SI FE
*SUB CLASS    2 UL 25 87
                LL 13 75
POP. =        2
POP.%=  50.00  AREA %=  58.74  AREA*XRAY %=  35.49  AVG XRAY CNT =      1404.
PARTICLES PER SQCM.= 1.16E 00  AVG.AREA = 1.86E 03  (SQ.UM.) SIGMA= 5.73E 02
============================================================================
MACRO CLASS   2 : FE
POP. =        2
POP.%=  14.29  AREA %=  14.98  AREA*XRAY %=  11.65  AVG XRAY CNT =       903.
PARTICLES PER SQCM.= 1.16E 00  AVG.AREA = 1.48E 03  (SQ.UM.) SIGMA= 3.10E 02
============================================================================
MACRO CLASS   3 : SI CU
                %S 37 63
POP. =        2
POP.%=  14.29  AREA %=  20.53  AREA*XRAY %=  12.39  AVG XRAY CNT =       702.
PARTICLES PER SQCM.= 1.16E 00  AVG.AREA = 2.03E 03  (SQ.UM.) SIGMA= 1.02E 03
----------------- SI CU
*SUB CLASS    1 UL 25 87
                LL 13 75
POP. =        1
POP.%=  50.00  AREA %=  32.15  AREA*XRAY %=  40.43  AVG XRAY CNT =       882.
PARTICLES PER SQCM.= 5.81E-01  AVG.AREA = 1.30E 03  (SQ.UM.) SIGMA= 0.00E 00
----------------- SI CU
*SUB CLASS    2 UL 50 62
                LL 38 50
POP. =        1
POP.%=  50.00  AREA %=  67.85  AREA*XRAY %=  59.57  AVG XRAY CNT =       616.
PARTICLES PER SQCM.= 5.81E-01  AVG.AREA = 2.75E 03  (SQ.UM.) SIGMA= 0.00E 00
============================================================================
MACRO CLASS   4 : SI
POP. =        4
POP.%=  28.57  AREA %=  26.59  AREA*XRAY %=   6.36  AVG XRAY CNT =       278.
PARTICLES PER SQCM.= 2.32E 00  AVG.AREA = 1.31E 03  (SQ.UM.) SIGMA= 1.10E 03
============================================================================
MACRO CLASS   5 : FE NI
                %S 84 16
POP. =        1
POP.%=   7.14  AREA %=   2.92  AREA*XRAY %=   4.60  AVG XRAY CNT =      1833.
PARTICLES PER SQCM.= 5.81E-01  AVG.AREA = 5.75E 02  (SQ.UM.) SIGMA= 0.00E 00
----------------- FE NI
*SUB CLASS    1 UL 87 25
                LL 75 13
POP. =        1
POP.%= 100.00  AREA %= 100.00  AREA*XRAY %= 100.00  AVG XRAY CNT =      1833.
PARTICLES PER SQCM.= 5.81E-01  AVG.AREA = 5.75E 02  (SQ.UM.) SIGMA= 0.00E 00
============================================================================
MACRO CLASS   S :
POP. =        1
POP.%=   7.14  AREA %=   2.46  AREA*XRAY %=   0.00  AVG XRAY CNT =         0.
PARTICLES PER SQCM.= 5.81E-01  AVG.AREA = 4.86E 02  (SQ.UM.) SIGMA= 0.00E 00
```

FIGURE 15

RELATIVE INTENSITY CHEMICAL CLASSIFICATION.

(Explanation of headings and terms on next page)

EXPLANATION OF TERMS FOR MACRO CLASS 1, FIGURE 15

SI FE - The particles in this class contain silicon and iron.

%S - The X-ray relative intensity in percent is given immediately below each elemental symbol, i.e., 11% Si and 89% Fe for the first macro class shown.

POP - The number of particles in the Macro class.

POP% - The percentage of the total particles in the 37 frames that fit into this class.

AREA% - Percentage of total area (37 frames) that belongs to this class.

AREA * XRAY% - Contribution of all the particles in the class to the total X-ray counts made (area weighted).

AVG XRAY CNT - Average X-ray counts for the class.

PARTICLES PER SQCM - Number of particles divided by the area analyzed in centimeters.

AVG. AREA - Average particle area for the class expressed in square micrometers.

SIGMA - The standard deviation of the distribution of the class area measurements.

The terms for the subclasses are the same except that POP%, AREA%, and AREA*XRAY% are percentages of the Macro Class instead of the percentages of all particles in the 37 frames.

The customized printout being used by RIA is shown in Figure 16.

SAMPLE ID: M140-A11679-NEW GD W/ I/OSUP US35 COVERAGE1 0735   17MAY83

NUMBER OF PARTICLES BY CHEMISTRY AND EQUIVALENT LENGTH (UM) RANGE

CHEMICAL CATEGORY \	< 4.00 E 1	< 6.00 E 1	< 2.00 E 2	< 4.00 E 2	< 6.00 E 2	> 6.00 E 2	/ TOTAL
Z < 9 (=F)	0	0	1	0	0	0	1
LINT/FIBERS	0	2	2	0	0	0	4
MET.FERROUS	0	1	6	0	0	0	7
MET NONFERR.	0	0	0	0	0	0	0
ABRASIVE	0	0	2	0	0	0	2
NON-ABRASIVE	0	0	0	0	0	0	0
MISC.MACRO	0	0	0	0	0	0	0
SUM UNKNOWN							0
TOTAL	0	3	11	0	0	0	14

FIGURE 16

RESULTS IN RIA FORMAT.

(Explanation of headings and terms on next page)

EXPLANATIONS OF TERMS AND HEADINGS IN FIGURE 16

SAMPLE ID: Followed by Gun Mount Type - Serial number - New or Rework Mount.

GD - Gridameter Mode. W/I/O Sup - unwanted tables and histograms suppressed DS35 - serial number of data storage disk.

Coverage - Type of run, time run started, and date of run.

Title of Table - Self explanatory (length measured in micrometers).

Chemical Category.

$Z < 9$ (=F) - Particles detected by backscattered electrons but whose X-rays were not detectable, that is, elements with an atomic number less than 9 (fluorine). These are usually organic materials.

LINT/FIBERS - Particles whose width to length ratio is less than 0.300 and whose iron X-rays are less than 40% of the total X-rays from that particle.

MET. FERROUS - Particles whose iron X-rays are more than 50% of the total X-rays from that particle.

MET. NON FERR. - Particles whose aluminum, chromium, nickel, copper, or zinc X-rays are greater than 40% of the total X-rays from that particle.

ABRASIVE - Particles whose magnesium and silicon, silicon and iron, silicon alone, or titanium X-rays are greater than 35% of the total X-rays from that particle.

NON ABRASIVE - Particles whose chlorine or molybdenum disulfide, chlorine, and phosphorous X-rays are greater than 40% of the total X-rays from that particle.

MISC. MACRO - Any particle which cannot be classified in any of the above will be put into this class.

SUM UNKNOWN - If all computer memory allocated for the collection of the classes above is used, additional particles found will be counted, but not classified. The total number of such particles will be given.

The lengths ranges for the columns are:

< 4.00 = Less than 40 micrometers E1

< 6.00 = 40 to 59.999 micrometers E1

< 2.00 = 60 to 199.99 micrometers E2

< 4.00 = 200 to 399.99 micrometers E2

< 6.00 = 400 to 599.99 micrometers E2

> 6.00 = 600 micrometers and larger E2

The particle described in Figure 14, because of its high iron content and length between 60 and 199 micrometers is one of the six in row 3 column 3.

3.0 RESULTS AND DISCUSSION

3.1 Optical Observations.

As mentioned previously, the LeMont programs allow all of the original data from counting runs to be saved on magnetic disks. The data can then be reexamined using a different format to gain additional information. All samples were counted first using the custom RIA format with the data save storage option. Then the data from each sample was rerun using the particle by particle format.

The optical observation made during the preparation of the filters for automated counting was cursory, looking only for larger (minimum size - 60 micrometers) metallic particles. A magnetic probe that normally is used to separate ferrous from non-ferrous or soft (non-abrasive) from hard (abrasive) particles was not used to preclude disruption of the filter surface before carbon coating.

Table VI is a summary of the optical observations versus the automated particle counts for metallic particles larger than 60 micrometers. Examination of the table reveals the expected general trend where the number of particles counted by the automated system increased as the number observed optically increased.

The automated system found more particles than had been counted optically in 60 percent of the samples, an equal number in 13 percent of the samples, and fewer particles in 27 percent of the samples. The greatest discrepancy was in sample M45, serial number 232 where the optical method found 5 particles and the automated none. The number of particles found by both methods in each of the M45 samples was so small that none of them are significant when compared with the criteria listed in Table I. Neither counting method would have failed one of the M45 samples.

For the other four types of mechanisms, the ratios of the number of particles found by the automated count to the optical count was, in general, similar for each mechanism, i.e., for the M1 - 2 to 1, the M140 - 5 to 1, the M174 - 10 to 1, and the M178 - 10 to 1. Why all of the ratios were not the same could not be explained. However, these data do show that if there are a significant number of particles in a sample, the automated system will detect more of them than the optical method.

Figure 17 is an optical photomicrograph of a typical sample (M178, SN 4984) prepared for SEM counting. A profusion of white fibers can be seen on this type of filter. The large number of fibers in the hydraulic fluid has hitherto not been noted. During routine quality assurance optical counting the fibers can not be distinguished from the fibers used to make up the filter paper (Figure 18).

Data obtained from optically counting lint in split samples from five M1 gun mounts on both types of filters are presented in Table VII. As

TABLE VI

Optical Observation Versus Automated Particle Counts
(Number of Metallic Particles Observed > 60 Micrometers)

Mechanism/Serial Number	Optical Metallic	SEM Ferrous	SEM Non-Ferrous
M1/014	5	2	10
015	5	13	4
016	1	0	0
017	7	4	5
018	10	6	156*
M45/288	0	3	0
229	0	1	0
230	2	0	1
231	1	0	0
232	5	0	0
M140/11679	0	8	2
11680	2	10	0
11681	2	0	1
11682	3	2	1
11683	1	4	1
M174/491	0	4	0
492	2	1	1
493	3	17	21
494	7	60	18
495	3	23	6
M178/4871U	0	0	0
4871L	3	0	0
4983U	1	0	0
4983L	10	8	0
4984U	2	14	5
4984L	0	23	2
5290U	0	1	1
5290L	0	0	0
5291U	3	1	31
5291L	0	1	19

*LeMont discriminator may have been improperly set.

FIGURE 17

PHOTOMICROGRAPH OF A NUCLEPORE POLYCARBONATE MEMBRANE FILTER, USED IN AUTOMATED PARTICLE COUNTS, WITH FIBERS FROM A HYDRAULIC FLUID. LIGHT MICROSCOPE VIEW AT 20X.

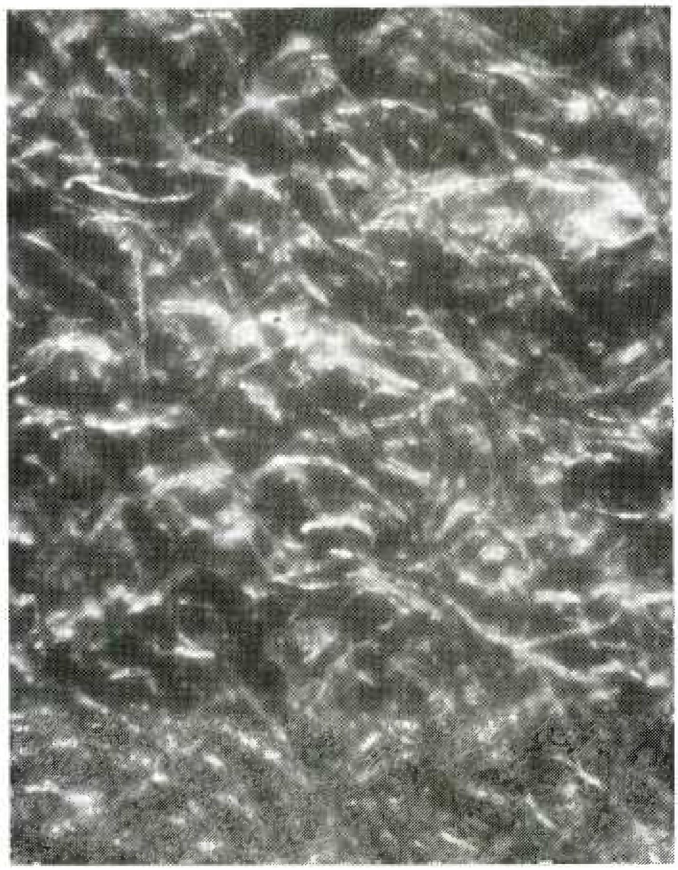


FIGURE 18

PHOTOMICROGRAPH OF A NEW OR UNUSED FIBROUS FILTER PAPER THAT IS USED IN OPTICAL COUNTING. LIGHT MICROSCOPE VIEW AT 50X.

TABLE VII

Lint Material from Split M1 Gun Mount Samples Counted Optically on Fiber Filter Papers and on Nuclepore Filters

SN	Number of Particles (Length Range in Micrometers)		Filter Type	Nonmetallic Lint and Fiber Results
	(100-600)	(>600)		
014	0	1	White Fiber	Fail
	120	27	Nuclepore*	Fail
015	0	0	White Fiber	Pass
	55	12	Nuclepore*	Fail
016	0	0	White Fiber	Pass
	25	5	Nuclepore*	Fail
017	0	0	White Fiber	Pass
	55	10	Nuclepore*	Fail
018	0	0	White Fiber	Pass
	35	5	Nuclepore*	Fail

*More than one filter was required for the sample on the nuclepore because of the large quantity of particulates.

expected from viewing Figures 17 and 18, a much greater quantity of lint is found when optically counting the Nuclepore filter than when optically counting the filter paper.

3.2 Samples Counted Automatically with SEM.

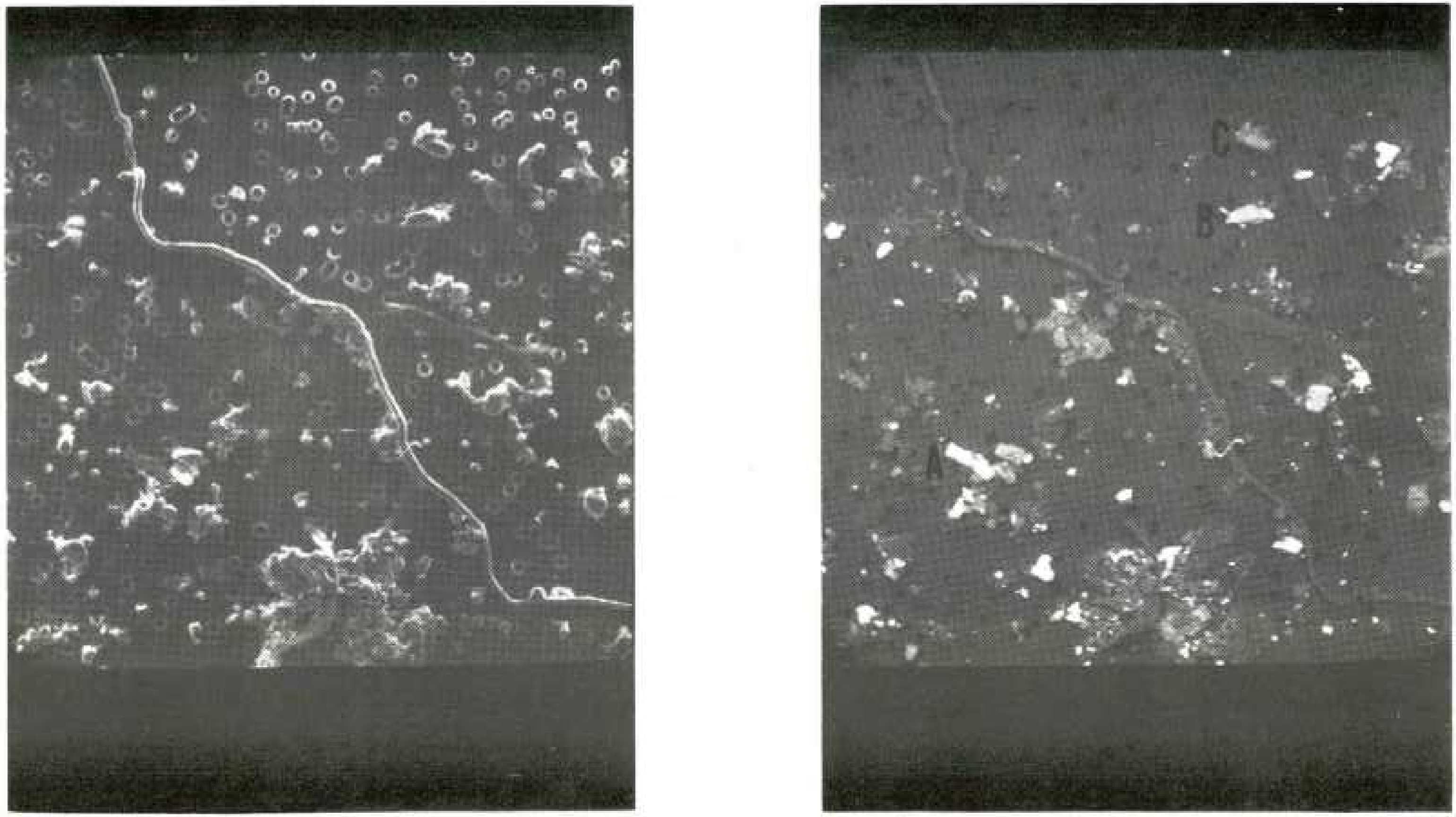
Figure 19 shows both a secondary electron and a backscattered electron micrograph of a single fiber on the sample mentioned in the previous paragraph. The fiber can easily be seen running from the upper left corner toward the lower right corner in (a) (secondary electrons), while in (b) (backscattered electrons), the fiber, although visible, is very similar in appearance to that of the filter substrate. The LeMont system relies on differences in intensity of the backscattered electron signal rather than secondary electrons to tell when it has found a particle to be examined. Consequently, this fiber would not be considered a particle because its signal intensity would be so similar to the background. Hence, few of the organic fibers are counted by the LeMont system; just as few have been counted in the past and few are currently counted optically.

The fiber counting problem can be resolved by the use of an optical microscope. Reference to Table I reveals that much larger sizes for fibers are allowed than sizes for the other categories. Examination of the Nuclepore filters prepared for SEM counting at low power in an optical microscope will allow fibers to be easily seen. If a sample has fibers larger than allowed, the mechanism can be failed without further work. If the sample passes the optical test for fiber content, SEM counting can proceed.

There are many particles in Figure 19(b) which would trigger the LeMont program. They are either white (in the photomicrograph) or a much lighter gray than the filter itself. EDS analysis shows that the white particle, A, is iron, the light gray particle, B, is a combination of calcium and sulfur, and the darker gray particle, C, is silicon. No X-rays were detected from the fiber showing that it is a typical organic compound composed of mainly carbon, hydrogen, and oxygen — light elements whose X-rays are not detectable by the RIA EDS. Figure 19(b) shows that heavier elements yield a more intense (hence whiter appearance) backscattered electron signal.

The LeMont programs used during this investigation define a fiber as any particle with a width to length ratio less than 0.300 and having any composition other than containing 40 or more percent iron. Consequently, metallic non-ferrous particles and non-metallic particles have been classified as lint and fiber, which accounts for most of the particles that were classified as lint and fiber, even though true organic lint and fiber is difficult to detect.

Table VIII is a comparison of pass or fail results between actual routine quality assurance optical particle counts and automated SEM (RIA format) counts; the counts were performed on duplicate split samples. The



(A) (B)

FIGURE 19

ORGANIC FIBER AS VIEWED BY 19(A) SECONDARY AND 19(B) BACKSCATTERED ELECTRONS. ON FIGURE 19(B) THE FOLLOWING PARTICLES ARE DESIGNATED: A — IRON CONTAINING PARTICLE, B — CALCIUM AND SULFUR CONTAINING PARTICLE, C — SILICON CONTAINING PARTICLE. MAGNIFICATION 120X.

TABLE VIII

Comparison of Pass or Fail Results from Samples Optically Counted (OC) and Automatically Counted (SEM)

M1			M45			M140		
SN	OC	SEM	SN	OC	SEM	SN	OC	SEM
014	Pass	Pass	228	Fail(3)	Pass	11679	Pass	Pass
015	Pass	Pass	229	Pass	Pass	11680	Pass	Pass
016	Pass	Pass	230	Fail(4)	Pass	11681	Pass	Pass
017	Fail(1)	Pass	231	Pass	Pass	11682	Pass	Pass
018	Fail(2)	Pass	232	Pass	Pass	11683	Pass	Pass

M174				M178			
SN	OC	SEM	Frame (5)	SN	OC	SEM	Frame
491-A (6)	Pass	Fail	26	4871U (7)	Fail (8)	Pass	
491-B		Fail	16	4871L	(9)	Pass	
492-A	Pass	Pass		4983U	Pass	Pass	
492-B		Fail	16	4983L	Fail	Fail	13
493-A	Pass	Fail	15	4984U	Pass	Fail	3
493-B		Fail	14	4984L	Pass	Fail	9
493-C		Fail	12	5290U	Pass	Pass	
494-A	Pass	Fail	7	5290L	Pass	Pass	
494-B		Fail	9	5291U	Pass	Fail	23
494-C		Fail	1	5291L	Pass	Fail	5
495-A	Pass	Fail	3				
495-B		Fail	5				

(1) Reason failed: One NONMETALLIC Particle > 600 micrometers.
(2) Reason failed: One MET. NONFERROUS Particle > 600 micrometers.
(3) Reason failed: One NonMetallic Particle > 400 micrometers.
(4) Reason failed: One MET. NONFERROUS Particle > 400 micrometers.
(5) All failures for the M174 and M178 were flagged when the program counted a total of more than two metallic ferrous particles greater than 40 micrometers. The count then automatically stopped in the frame indicated.
(6) More than one filter required for sample because of large quantity of particles.
(7) U and L indicate Upper or Lower cylinders.
(8) Reason failed: One MET. NONFERROUS Particle > 400 micrometers.
(9) Not counted when upper has already failed.

large differences in the pass-fail rate between the M1-M45-M140 groups and the M174-M178 group, where in the latter group the SEM rejected more samples, can be explained by the differences in particle sizes allowed in Table I and by the differences in counting methods.

Many of the samples failed by the SEM, which had passed the optical count, were caused by particles in the lower portion of the 40 to 50 micrometer range. A human being can easily skip over these smaller particles while making a subjective, optical count. The automated system, however, being objective measures every particle it encounters. Even though the automated system does not look at the entire surface of the filter (which will be discussed later in this report), it detected enough of the just above 40 micrometer particles to fail mechanisms which the optical method has passed. If automated particle counting replaces the optical method, the oil contamination criteria for the M174-M178 group will need to be changed to reflect the automated system's greater accuracy.

The data from the 30 Relative Intensity Chemical Classification summaries generated during the particle by particle printout for each mechanism were consolidated into groups based on the predominant element (over 50 percent relative intensity). The number of particles in each group was listed for each mechanism, Table IX. The number of particles has no relationship to particle size, just to composition. The summary includes <u>all</u> particles that were seen by the SEM. Possible sources for particles in the various groups are listed below:

a. No X-rays - Organic compounds such as rubber, plastics, or fibers which because of their topography triggered the LeMont program, but did not emit detectable X-rays.

b. Iron - Any of the many steel parts of the mechanism.

c. Copper - The various bronzes used in the mechanisms.

d. Copper-Zinc - Brass components and fittings.

e. Silicon - Silicon dioxide in abrasive compounds or dust.

f. Tin - Anti-friction metal.

g. Zinc - Zinc phosphate coatings.

h. Titanium - Titanium oxide in paint.

i. Aluminum - Aluminum oxide abrasives, aluminum components.

j. Cadmium - Cadmium plated components.

k. Chlorine - Chlorinated organic compounds, sodium chloride (often found in dust).

TABLE IX

Particle Distribution in Major Elemental Groups

Mechanism	No. X-Rays	Iron	Copper	Copper and Zinc	Silicon	Tin	Zinc	Titanium	Aluminum	Cadmium	Chlorine	Molybdenum and Sulfur	Nickel
M1 SN 014		11	43										
015		22	18		1	1							
016		1	1										
017	1	9	6					1					
018	28	7	217										
M45 SN 228	8	93	12	23	8	2		4				2	
229	1	2						2					
230				1									
231	No Particles Detected												
232	No Particles Detected												
M140 SN 11679	12	23	3		3								
11680		106											2
11681		3	3							1			
11682	2	10							2	1	1		
11683	1	6	2		1		1						
M174 491	4	23	5	10	22	4	1						
492	4	2	4	4	21	1	3	1					
493	8	107	55	70		24	11	1	11	2			
494	14	195	16	97	1	7	1		1	16			
495	5	189	28	42	2	15		7		3			
M178 SN 4871U			6										
4983U	1		3										
4984U		28	11			2				2			
5290U		2	7		1		4						
5291U			22		9					2			
SN 4871L		1											
4983L	1	9	5				1	1				1	
4984L	5	33	5	2						3			
5290L		1	3	1	8		2					5	
5291L		7	101	16									

l. MS - Molybdenum disulphide lubricant.

m. Nickel - Nickel plating.

The significance of Table IX is that never before has data been available in this detail. The particle by particle printout, while not practical for routine counts, can always be made available from the data saved on magnetic disk. These data which are objective, not subjective observations, can be used to establish any type of data base desired such as "What are the major alloy types found in the Hydraulic Fluid?" or "What are the actual sizes and shapes of the various abrasive particles found?" This type of information will be most useful to engineers developing new recoil mechanisms or to those who must specify quality assurance criteria.

That iron and copper base materials are the most prevalent in all of the mechanisms is to be expected. The number of particles in the other elemental groups varies between the types of mechanism. Also note that, as would be expected, there is more variety of particles as the total number of particles increase. Several factors are involved in the variation in the total number of particles found per mechanism. First, and most obvious, some mechanisms are cleaner than others. Second, the precision with which the various electronic parameters can be set on the LeMont and ETEC instruments varies. Two opposing factors are involved in the present configuration of the instrumentation.

One Factor: To make the electronic set up as objective as possible, the intensity of the backscattered signal from the Nuclepore filter (Figure 20A) was kept at a specified spacing (5 millimeters) from the intensity of the holes in the filter when examined in the viewing screen in the wave form mode - see Figure 20B. The ETEC backscatter detector is excellent for making photomicrographs, where time is not a factor, and scan lines can move slowly enough to insure an adequate signal. In the higher speed environment required in the LeMont system, the only way to increase the initial backscatter signal is to increase the SEM electron beam intensity. Unfortunately, increasing the beam intensity also increases the X-ray yield from the sample being studied. The X-rays can become so intense that the X-ray detector will become saturated and cease to function. A compromise had to be made between the two parameters and the specimen current was kept at 1x10E-09 ampere to maintain an optimum X-ray yield. The amount of backscatter electron amplification required to produce an adequate backscattered electron signal for the LeMont also amplified the ambient electronic noise which made the setting of the LeMont threshold for particle detection difficult to reproduce for each filter - see Figure 20C. Some variation between mechanisms in the number of particles detected was caused by the problems involved with this high signal to noise ratio. The problem will be eliminated with the eventual acquisition of a faster, more sensitive backscatter electron detector.

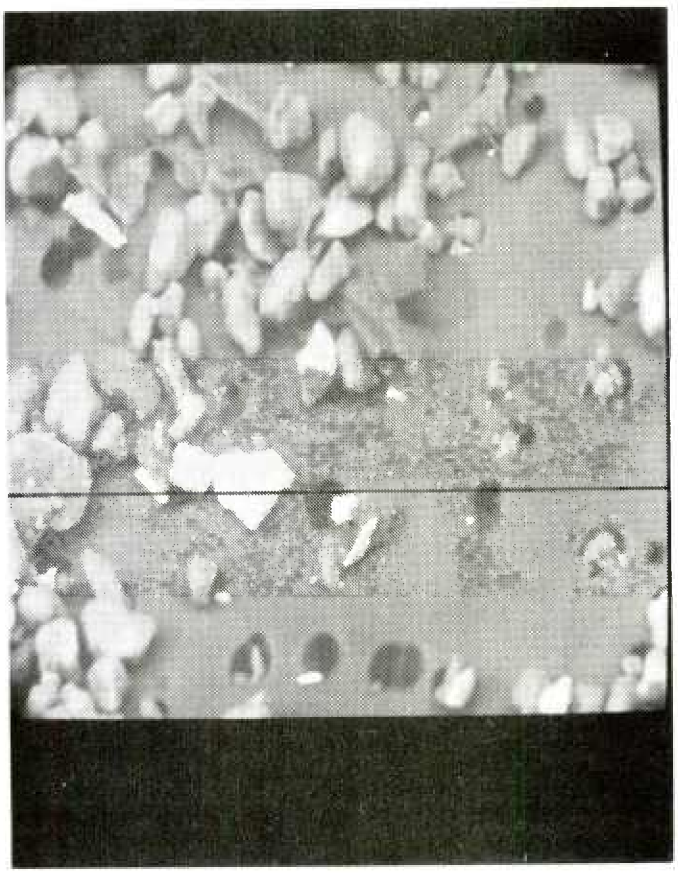


FIGURE 20(A)

PHOTOMICROGRAPH WITH A SCAN LINE THROUGH BRIGHT PARTICLES AND FILTER HOLES. MAGNIFICATION 400X.

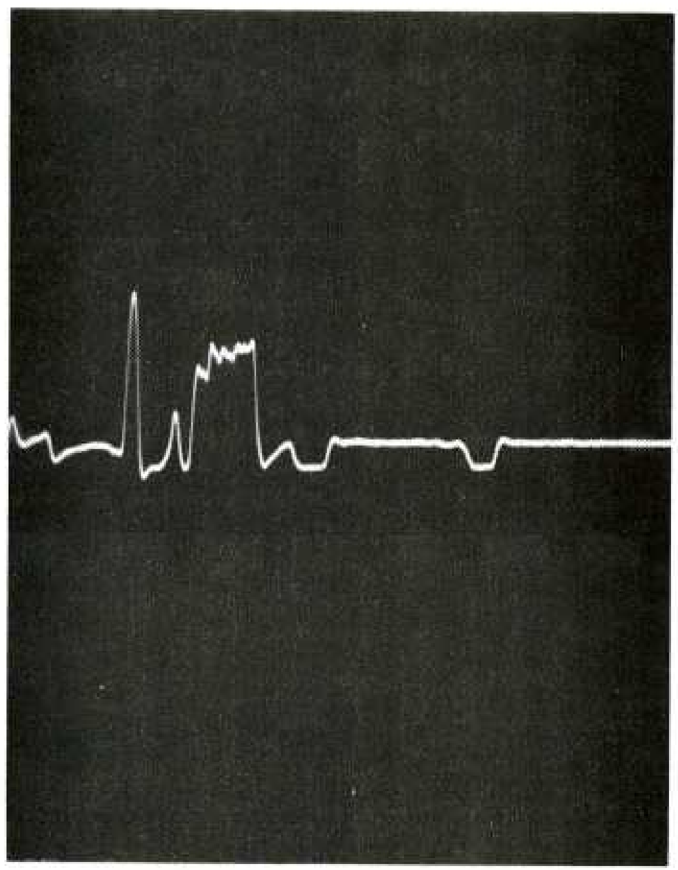


FIGURE 20(B)

WAVE FORM OF SCAN LINE SHOWN IN FIGURE 20(A) (HIGH SPECIMEN CURRENT). PEAKS CORRESPOND TO BRIGHT AREAS, DEPRESSIONS TO FILTER HOLES.

WAVEFORM OF SCAN LINE SHOWN IN FIGURE 20(A) (1x10E-09 AMPERE SPECIMEN CURRENT). NOTE INDISTINCT WAVEFORM COMPARED TO FIGURE 20(B).

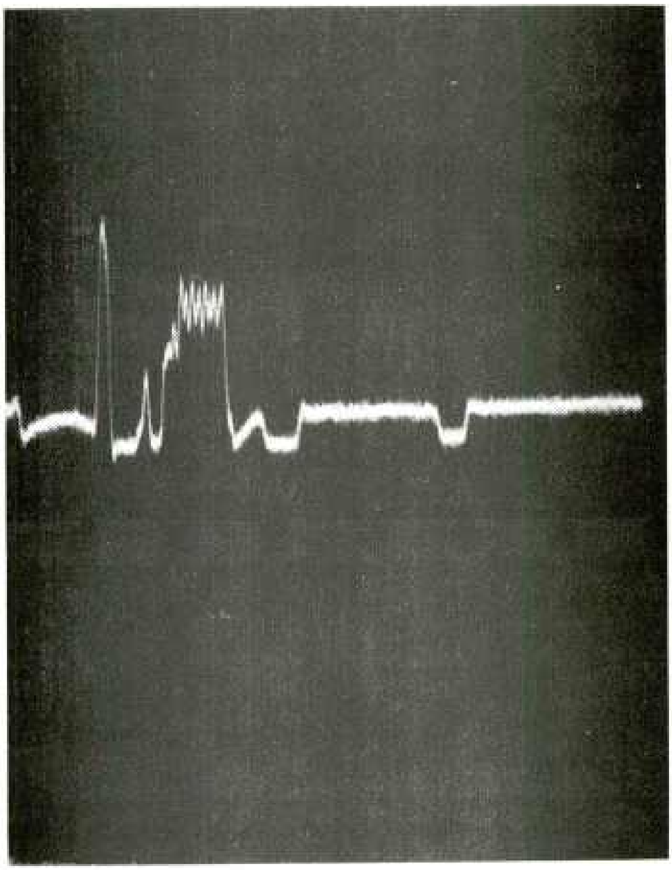


FIGURE 20(C)

WAVEFORM OF SCAN LINE SHOWN IN FIGURE 20(A) (1x10E-09 AMPERE SPECIMEN CURRENT). NOTE INDISTINCT WAVEFORM COMPARED TO FIGURE 20(B).

The Other Factor: The manner in which the LeMont program looks at the area to be counted could cause differences in the number of particles detected. During sample preparation, the base and O-ring of the Swin-Lock holder (Figure 12) form a 23 millimeter diameter mask on the filter which results in a usable area of 415 square millimeters. The program uses the diameter of the sample and the stipulated magnification (40x) to determine the size and number of frames for a complete count.

In the RIA system, as previously stated, 40x magnification yields a frame size of 2.25 millimeters square. The program calculates the length of the frame diagonal (in this case 3.18mm) and uses this distance as the increment for each x translation of the stage. Moving at least the length of the frame diagonal insures that as the sample is rotated, no part of a previously counted frame will be included in the current or future frames. The program then divides the radius of the sample by the diagonal length to determine the number of rings to be used. In this case 11.5 divided by 3.18 = 3.62 which means that only 3 complete rings can be looked at within the sample area. The calculations resulted in the program looking at 37 frames of 5.06 square millimeters each for a total of 187 square millimeters; only 45 percent of the 415 millimeter area of the sample. Since there are only three x translations of 3.18 mm each, the diameter of the area actually counted is expressed in the following equation: $[(3 \times 3.18\text{mm})] + 1.25\text{mm} \times 2 = 21.6\text{mm}$

This difference between the available sample diameter of 23mm and the diameter of the sample actually counted, 21.6mm, insures that if the filter area is not accurately centered on the sample, the outside circle of frames will not intersect the outer diameter of the filter area.

The filtering procedure was developed to insure that particles were spread over the surface of the filter in a random and even distribution. Despite the fact that the LeMont program looks at only 45 percent of the surface, the sampling pattern and the random distribution insure that a representative portion of the particles will be examined.

The actual time required to count a sample on the automated system depends, of course, on the number of particles found and examined. The average time taken to examine a sample which yielded no particles was 25 minutes. This was the time required for the sample to be rotated, translated, each frame examined, and the results printed out. As the system is now configured, the printer is the slowest component and while each results is being printed, no other function (motion or counting) can occur. The average time to size, determine composition and print out the data for a particle was 11 seconds.

As mentioned in the Test Plan, the M140 sample, SN 11679, was counted twelve times. Before each count, the initial position of the sample was rotated 30 degrees so that during the twelve counts the sample was observed from twelve different aspects differing by 30 degrees each.

Results of the twelve counts are given in Table X. Note that no particles longer than 200 micrometers were found. The variations in the number of particles found in each count can be attributed to the difficulty in reproducing the electronic parameters mentioned above and the rotation of the sample, i.e., some of the particles would not be in an area being scanned.

Particle by particle printouts were prepared from each of the twelve counts. By comparing size, composition, and the ring that they appeared in, it was possible to identify and track a total of 24 particles that were 80 micrometers or more in length. The average size of each particle was calculated and for those seen more than twice the coeffieient of variation was also calculated. Table XI is a summary of the data for these particles giving composition, number of times found, the ring in which it was found, and their lengths and widths. Lengths varied from 86.45 to 179.46 micrometers; widths from 8.80 to 32.55 micrometers. Over half of the particles were either of iron or iron-silicon composition, five showed more than 99 percent relative intensity for silicon only and the rest were copper base materials or no X-rays (organic). Particles with silicon content greater than 99 percent could be organic compounds containing silicon, but whose other elements did not emit detectable X-rays. These sizes and length to width ratios indicate that they were not abrasives.

The average coefficients of variation give an indication of how reproducible the sizing function of the LeMont system is. That the coefficient for both length and width is less than ten percent, speaks well of the capabilities of the system.

TABLE X

Size Distribution of Particles Detected in Repeatability Study
(Size Ranges in Micrometers)

Run Number	No. Particles per Size Range <40	40-60	60-200	200-400	400-600	>600	Total Particles	No. Particles in 40-200 Micrometers Range
1	0	3	11	0	0	0	14	14
2	5	4	16	0	0	0	25	20
3	0	5	8	0	0	0	13	13
4	0	2	11	0	0	0	13	13
5	2	3	11	0	0	0	16	14
6	2	2	18	0	0	0	22	20
7	3	3	14	0	0	0	20	17
8	2	4	13	0	0	0	19	17
9	3	6	15	0	0	0	24	21
10	0	1	14	0	0	0	15	15
11	3	8	13	0	0	0	24	21
12	1	1	15	0	0	0	17	16

TABLE XI

Data Summary of 24 Particles Detected in Repeatability Study

Composition	Number of Times Found	Found In Ring Number	Length (Average in micrometers) $\overline{X}$	Length V(%)	Width $\overline{X}$	Width V(%)
Fe 99	4	3	93.95	9.4	25.14	10.1
Fe 99	3	3	113.97	4.2	15.69	4.2
Fe 99	3	2	93.58	2.5	31.08	6.3
Fe 99	2	4	158.68		12.06	
Fe 99	2	2	87.71		18.88	
Fe 99	2	2	97.18		21.10	
Fe-93, Si-7	3	4	88.51	8.8	19.48	10.8
Fe-92, Si-8	3	4	179.17	3.0	12.08	5.3
	3	2	88.89	8.9	18.87	10.3
Fe-90, Si-10	2	4	89.52		16.58	
Fe-89, Si-11	3	3	86.45	8.8	27.92	9.6
Fe-84, Si-16	2	2	97.75		20.78	
Fe-80, Si-20	3	2	96.13	5.0	32.55	3.0
Si 99	4	4	147.50	7.2	19.39	8.5
Si 99	3	4	95.56	12.2	14.36	13.3
Si 99	2	4	91.46		15.88	
Si 99	2	3	87.93		8.80	
Si 99	2	3	128.28		31.88	
Cu-73, Si-27	4	3	90.50	8.2	14.33	13.9
Cu-56, Si-44	5	4	96.31	7.1	29.15	6.4
No X-rays	5	3	129.38	7.9	30.34	8.0
No X-rays	3	3	103.41	15.5	10.43	14.9
No X-rays	3	2	103.99	7.3	13.49	15.2
No X-rays	2	2	179.46		12.32	
Average of V(%)				7.73		9.32

4.0 CONCLUSIONS AND RECOMMENDATIONS

The LeMont scientific particle counting instrumentation and computer programs are operational and perform as specified in the original contracts.

The complete automated counting system (LeMont and the RIA Scanning Electron Microscope) can perform particle counts and has done so for a limited number of samples (five each) for the various recoil mechanisms produced at Rock Island Arsenal. Results show that the system, being entirely objective, gives more precise measurement and accurate characterization of the particles than the subjective and error prone optical method.

Automated particle counting should replace the currently used optical technique when a dedicated, specifically configured system is procured.

Particle size and characterization requirements currently in force do not make full use of the information available from the automated system.

The presently configured system should continue to be used for expanding the data base on particles found in hydraulic recoil mechanisms. One mechanism, the M1, should be studied in more detail. The increased sensitivity of the automated system should be used to study such things as reducing the hydraulic fluid sample size from 1 pint to 50 milliliters (to reduce sample preparation time), determining specifically what types and sizes of particles are detrimental to the mechanism, and revising particle count requirements to have more realistic and meaningful limits.

A more versatile, sensitive backscattered electron detector which would improve image signals should be procured for the present system. Such a detector would enhance the capabilities of the SEM for studies other than particle counting. The detector would be particularly useful in metallurgical studies.

A smaller, faster carbon coating apparatus which would significantly reduce sample preparation time should be obtained.

A dedicated automated particle counting system should be procured. The system should be composed of three major components: a scanning electron microscope, an energy dispersive x-ray spectrometer, and a particle counting system which will use information obtained from the first two instruments to perform particle counts.

Specific requirements are as follows:

a. The SEM should have a large specimen chamber to accommodate multiple or larger sized samples. Sample positioning controls should be motorized for computer operation. Focus and specimen current should be monitored and automatically held constant during unattended operation. All functions related to particle counting should be in digital format for ease of controlling by computer.

b. The EDS detector should have 155 electron volt resolution and be capable of detecting elements as light as sodium. The multichannel analyzer should have a resolution of 10 electron volts per channel. Again all functions should be digitized for ease of control by computer.

c. The particle counting system should have the hardware needed for processing signals received from the SEM and EDS for transmission to the computer. The programs for the computer (as well as all of the above) should be the latest state of the art. Programs should be provided to handle the specialized Rock Island Arsenal particle counts.

Two new techniques should be considered for possible inclusion in the dedicated system. First, use a speciman chamber which operates at a higher pressure than the rest of the electron column and which has been reported to allow examination of non-conductive samples without requiring the application of a conductive coating. [1] Special care should be taken to ascertain whether such a chamber could be used with an energy dispersive spectrometer. Second, incorporate a recently announced technique of using a computer to analyze backscattered electron intensities for determination of composition of low average atomic weight materials and oxides. [2] This capability would enable the system to identify more precisely organic matter and abrasives which are mainly oxides of silicon and aluminum.

[1] "SEM Examination of Nonconducting Specimens," American Laboratory, April 1983, pages 56-61.

[2] "Fast Compositional Analysis on an SEM", Industrial Research and Development, Jun 1983, page 123.

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AUTOMATED IDENTIFICATION, SIZING AND COUNTING OF PARTICULATE CONTAMINATION FOUND IN HYDRAULIC RECOIL SYSTEMS
by W. D. McHenry and L. A. Post

Report EN-84-12, August 1984, 57 p, incl. figures and tables. Unclassified report.

The primary project objective, to develop requirements and procedures for the automated identification, sizing, and counting of particulate contamination found in the hydraulic recoil systems, has been successfully completed. The automated system can provide meaningful, accurate data in various formats and a dedicated Automated Particle Analysis system has the potential to provide results quickly to meet production needs on an unattended operating basis.

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2. Scanning Electron Microscope
3. Secondary Electrons
4. Backscattered Electrons
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6. Energy Dispersive Spectrometer
7. LeMont Image Analysis System (B-10)
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2. Scanning Electron Microscope
3. Secondary Electrons
4. Backscattered Electrons
5. Characteristic X-rays
6. Energy Dispersive Spectrometer
7. LeMont Image Analysis System (B-10)
8. Automated Particle Counting System
9. Data Save
10. Chemical Definition File
11. Diameter
12. Gridameter
13. Relative Intensity Classification

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AUTOMATED IDENTIFICATION, SIZING AND COUNTING OF PARTICULATE CONTAMINATION FOUND IN HYDRAULIC RECOIL SYSTEMS
by W. D. McHenry and L. A. Post

Report EN-84-12, August 1984, 57 p, incl. figures and tables. Unclassified report.

The primary project objective, to develop requirements and procedures for the automated identification, sizing, and counting of particulate contamination found in the hydraulic recoil systems, has been successfully completed. The automated system can provide meaningful, accurate data in various formats and a dedicated Automated Particle Analysis system has the potential to provide results quickly to meet production needs on an unattended operating basis.

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